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AD-A205 467

Report 2473

Corrosion and Lubricity Testing of Nonflammable USAF Formulated CTFE Base Hydraulic Fluid

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Report Date: September 1988

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SECURITY CLASSIFICATION OF THIS PAGE

| REPORT DOCUMENTATION PAGE | | | | Form Approved OMB No. 0704-0188 |
|---|--|---|--------------------------------|------------------------------------|
| 1a. REPORT SECURITY CLASSIFICATION Unclassified | | 1b. RESTRICTIVE MARKINGS None | | |
| 2a. SECURITY CLASSIFICATION AUTHORITY | | 3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited. | | |
| 2b. DECLASSIFICATION/DOWNGRADING SCHEDULE | | | | |
| 4. PERFORMING ORGANIZATION REPORT NUMBER(S) No. 2473 | | 5. MONITORING ORGANIZATION REPORT NUMBER(S) | | |
| 6a. NAME OF PERFORMING ORGANIZATION Belvoir RD&E Center Materials, Fuels & Lubricants Dir. | 6b. OFFICE SYMBOL (If applicable) STRBE-VF | 7a. NAME OF MONITORING ORGANIZATION | | |
| 6c. ADDRESS (City, State, and ZIP Code) Fuels & Lubricants Div. Fort Belvoir, VA 22060-5606 | | 7b. ADDRESS (City, State, and ZIP Code) | | |
| 8a. NAME OF FUNDING/SPONSORING ORGANIZATION | 8b. OFFICE SYMBOL (If applicable) | 9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER | | |
| 8c. ADDRESS (City, State, and ZIP Code) | | 10. SOURCE OF FUNDING NUMBERS | | |
| | | PROGRAM ELEMENT NO. 627.33 | PROJECT NO. AH20 | TASK NO. VLT |
| | | WORK UNIT ACCESSION NO. | | |
| 11. TITLE (Include Security Classification) Corrosion and Lubricity Testing of Nonflammable USAF Formulated CTFE Base Hydraulic Fluid (U) | | | | |
| 12. PERSONAL AUTHOR(S) Jane Scott Smith | | | | |
| 13a. TYPE OF REPORT Final | 13b. TIME COVERED FROM May 86 TO Nov 87 | 14. DATE OF REPORT (Year, Month, Day) September 1988 | | 15. PAGE COUNT 20 |
| 16. SUPPLEMENTARY NOTATION | | | | |
| 17. COSATI CODES | | 18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number) Chlorotrifluoroethylene (CTFE), Nonflammable Hydraulic Fluid (NFHF), Hydraulic Fluid, Halocarbons, Corrosion, Wear, Lubricity, FRH, MIL-H-46170 | | |
| 19. ABSTRACT (Continue on reverse if necessary and identify by block number) The objective of this study was to evaluate the antiwear and anticorrosion properties of the US Air Force's latest nonflammable hydraulic fluid formulation, a chlorotrifluoroethylene oligomer containing an antiwear additive and an anticorrosion additive. This formulation, referred to as NFHF, is stable to 135°C. The NFHF was tested according to ASTM D1472 and D1748, the Federal Test Standard 791C Methods 5322.1 and 5308.6, and the corrosion rate evaluation procedure (CREP). The data contained in this report is compared with the chlorotrifluoroethylene with no additives and the present fire resistant hydraulic fluid military specification MIL-H-46170. The antiwear and anticorrosion properties of the formulated NFHF improved over the base fluid with no additives. (Key words: Fire safety, Corrosion inhibition, fire resistant hydraulic fluid.) | | | | |
| 20. DISTRIBUTION/AVAILABILITY OF ABSTRACT <input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input checked="" type="checkbox"/> SAME AS REPORT <input type="checkbox"/> DTIC USERS | | 21. ABSTRACT SECURITY CLASSIFICATION Unclassified | | |
| 22a. NAME OF RESPONSIBLE INDIVIDUAL Jane Scott Smith | | 22b. TELEPHONE (Include Area Code) 703-664-4594 | 22c. Office Symbol STRBE-VF | |

PREFACE

The purpose of this report is to evaluate the lubricity (i.e., antiwear characteristics) and corrosion preventative properties of nonflammable hydraulic fluid (NFHF) using the following methods:

- ASTM D1472, *Wear Preventative Characteristics of Lubricating Fluids*
- Federal Test Standard 791C, Method 5308.6, *Corrosiveness and Oxidation Stability of Light Oils (Metal Strip)*
- Federal Test Standard 791C, Method 5322.1, *Corrosiveness of Oil on a Bimetallic Couple*
- ASTM D1748, *Rust Protection by Metal Preservatives in the Humidity Cabinet*
- Corrosion Rate Evaluation Procedure (CREP).

The results are compared with the base chlorotrifluoroethylene (CTFE) oligimer which represents a baseline, and the fire resistant hydraulic fluid, MIL-H-46170, to determine how the NFHF compares in these specific performance areas. In addition, specification requirements for antiwear and corrosion protection of NFHF may be established from this evaluation should this fluid be adopted for future applications that mandate a nonflammable hydraulic fluid.

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| Dist | Avail and/or Special |
| A-1 | |



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SECTION I. BACKGROUND

Petroleum-based hydraulic fluids have been a major contributor to fire-caused destruction of military equipment. Since the 1970s, the US Army, Air Force, and Navy have researched the development of hydraulic fluids that would increase fire protection. As a result of this effort, the Air Force developed MIL-H-83282, a synthetic hydrocarbon base fluid possessing superior fire resistance properties over its MIL-H-5606 (OHA) hydraulic fluid. Based on the Air Force's progress and the urgency for an improved fluid following the 1973-74 Middle East conflicts, the Army developed MIL-H-46170 fire resistant hydraulic (FRH) fluid to replace its MIL-H-6083 (OHT). FRH fluid is essentially a rust inhibited version of MIL-H-83282. The 1974 adoption of the FRH fluid was recognized as an interim solution. While the increased flash point and improved resistance to ignition and flame propagation properties of FRH fluid equate to an increase in safety over OHT in noncombat situations (service and training), its value in combat situations was felt to be marginal at best due to the extremely high temperatures encountered from direct fire and penetrating rounds.¹ Although fire resistant hydraulic fluid was desirable, the ultimate goal was an entirely nonflammable hydraulic fluid.

The Army and Air Force tested phosphate esters, silicones, chlorofluorocarbon, fluoroalkylether, and other classes of compounds for flammability as potential candidate fluids. The chlorofluorocarbon, specifically the chlorotrifluoroethylene (CTFE) oligimer, was ultimately selected as the candidate nonflammable hydraulic fluid.² CTFE fluid is almost double the density of the current petroleum-based fluid (i.e., MIL-H-6083) and incompatible with most elastomeric materials used in existing hydraulic systems. The Army originally attempted to formulate a CTFE base fluid compatible with elastomeric seals used in existing hydraulic systems.^{3, 4} It was realized during this effort that implementation of a CTFE base hydraulic fluid in existing hydraulic systems would not only require replacing all the elastomers due to incompatibility problems, but also modifying other hardware due to the high specific gravity. Therefore, the development of a nonflammable hydraulic fluid continued for eventual implementation within future armored vehicle systems because of the anticipated retrofit costs.

In 1985, the Army and Air Force established a formal cooperative program⁵ to coordinate their efforts in developing the CTFE base hydraulic fluid by resolving the corrosivity and low lubricity characteristics of this class of fluids. Additives were used to improve the characteristics of the base fluid. A formulation of the CTFE base hydraulic fluid, developed by the Air Force, used a special grade rust inhibitor and a proprietary lubricity additive, both thermally stable up to at least 135°C. The Air Force conducted field performance studies on a flameproof hydraulic brake system (FHBS) using the formulated CTFE base hydraulic fluid. After 6 months of testing, results indicated "no corrosion was found on any of the metal components. No scoring was found on any of the sliding metal components."⁶ This formulated CTFE base hydraulic fluid with rust inhibitor and lubricity additive stable to 135°C is referred to as *nonflammable hydraulic fluid (NFHF)* in this report.

SECTION II. TEST DETAILS

ASTM D4172 METHOD, *Wear Preventative Characteristics of Lubricating Fluids*

This method used the standard Roxana four-ball wear test machine. One rotating top ball is loaded against three stationary lower balls immersed in the test fluid. Test conditions were 10, 15, and 40kg loads at 1200 rpm at 75°C for 60 minutes. The wear preventative characteristics or lubricity of the fluid are determined by measuring the scars on each of the three stationary balls. The scar measurements is listed in Table 1. The only modification on the method was the addition of the 10kg load data in order that comparisons may be made with the Air Force's data. The NFHF under the 10 and 15kg load produced round scars. The 40kg load produced an elliptical scar.

FEDERAL TEST STANDARD 791C METHOD 5308.6, *Corrosiveness and Oxidation Stability of Light Oils (Metal Strip)*

This method evaluated the corrosive effects of an oil on five compositionally different metal coupons listed in Tables 2a and 2b. The five metal coupons were polished, cleaned, weighed, tied together, and submerged in a specific quantity of fluid for 168 hours at 121.1°C with a constant air flow through the fluid. Next, the coupons were carefully removed and cleaned. The percent evaporation, viscosity (at 38°C) of the remaining fluid, and the weight change of each metal was determined. Because NFHF degraded the method-prescribed cotton to hold the metal coupons together, nylon was used in place of cotton. After testing, the clear colorless nylon turned a burnt-red color indicating absorption of the anticorrosion additive. Therefore, the nylon was replaced by teflon thread made from teflon tape, and no complications resulted from its use. Although all data is provided in Tables 2a and 2b, the averages are based only on the data produced with the teflon thread modification.

FEDERAL TEST STANDARD 791C METHOD 5322.1, *Corrosiveness and Oil on a Bimetallic Couple*

This method determined the chemical or electrochemical effect of NFHF on a bimetallic couple (Table 3). The end of a steel disk was polished, cleaned, and coated with the test fluid, and then a brass clip was fitted to the fluid-coated surface. The assembly was stored in a humidity chamber at 26.7°C and 50% humidity (maintained with a saturated solution of magnesium nitrate hexahydrate) for 10 days. After 10 days, the brass clip was removed, and the steel disk was cleaned and inspected for corrosion at the site of the brass clip. This test method is generally completed in triplicate. The fluid tested fails if:

- More than one of the steel disks shows corrosion in the form of staining or pitting at the site of the clip, or
- Two disks show no corrosion and the third disk has more than three pits.

In order to increase the data statistics, the test was performed on 3 to 20 disks at a time; therefore, if more than a third of the disks had any form of corrosion, the fluid failed.

ASTM D1748 METHOD, *Rust Protection by Metal Preservatives in the Humidity Cabinet*

This method evaluated the rust preventative properties of a test oil on a steel surface under conditions of high humidity for an extended period of time. The specially prepared polished or sandblasted steel panels were dipped in the test oil and suspended in a humidity cabinet at 48.9°C for 100 hours. The panels were then removed, cleaned, and examined for rust spots or dots. If a rust dot was larger than 1mm or there were more than four dots in the specified areas, the steel panel failed. It is required that the panels be run in multiples of three. The plates referred to as *sandblasted* were actually blasted with glass beads due to the unavailability of the 120 grit sand at the time of the test (Table 4). It was observed that the results correlated with drying time when testing 12 panels at one time with NFHF. The longer the panel dried (within the ASTM drying time limits), the more likely it was to fail. Because of this observation, the number of panels tested with NFHF was reduced to three, eliminating the drying time variable. The results did not change.

CORROSION RATE EVALUATION PROCEDURE (CREP)

This Air Force-proposed procedure was used as a screening procedure to replace the ASTM D1748 method³ because it required less equipment, panel preparation, and time. NFHF was tested by the CREP to establish the criteria in the event the test is adopted. (Note: The CREP and apparatus are described in Report No. AFWAL-TR-81-4028, Part II.⁷) Two steel plates, one polished and one sandblasted, were cleaned in hot toluene followed by hot acetone. The test was performed in a sealed glass humidity chamber with a condenser, thermometer, constant air flow, two stoppers with hooks, and 100ml of distilled water in the bottom. The glass chamber was heated to 100°C and allowed to equilibrate for 1 hour with an air flow of 45 ml/min. The steel plates were weighed, dipped in the test fluid, hung up to dry, then suspended from the hooks on the stoppers in the glass chamber for exactly 1 hour. Next, the steel plates were cleaned and reweighed, and the weight change was recorded (Table 5). Although the procedure recommended the use of acetate buffer pH 4.7 instead of distilled water, the conditions were found to be too harsh. At this writing, no definite pass/fail criteria has been established for this proposed test procedure.

SECTION III. TEST RESULTS

The four-ball wear test indicated increased lubricity of the NFHF over the CTFE base fluid for the 10 and 40kg loads (Tables 1 and 6). The 10kg load produced a 0.50mm scar with NFHF, an improvement over the 0.81mm scar produced with CTFE base fluid; however, both exceeded the 0.25mm scar produced by the MIL-H-46170 fire resistant fluid. The 15kg maximum load limit for MIL-H-46170 is 0.30mm and compared unfavorably with the larger wear scars of 0.46mm produced by the NFHF and the 0.38mm scar produced by the CTFE base fluid. The wear scar for the 40kg load improved from an average 1.72mm for the CTFE base fluid to an average of 0.59mm for the NFHF. The wear for NFHF was significantly reduced in comparison to the CTFE base fluid and was below the maximum wear scar limit for MIL-H-46170 of 0.65mm for 40kg. The overall lubricity of the NFHF was definitely improved over the CTFE base fluid with increased loading, but did not perform as well as the fire resistant hydraulic fluid, MIL-H-46170, at the decreased loads.

The Federal Test Standard Method 5308.6 results indicated that the metal coupons tested in the corrosion inhibited NFHF experienced a weight change greater than that of the CTFE base fluid; however, the results were well within the limits of MIL-H-46170. The copper was tarnished, but did not exceed the No. 2 of the ASTM copper corrosion standards (ASTM D130). There was no evidence of pitting. The cadmium-plated steel showed neither pitting nor visible corrosion, although it was "dull." The steel, magnesium alloy, and aluminum alloy showed no visible changes. All of the observations were within the limits of the MIL-H-46170. The viscosity of the fluid before and after the testing did not change (Table 2b). The total acid number (TAN) of the NFHF increased, exceeding the limits of change specified in MIL-H-46170 (Table 2a). The significance of this will be determined in follow-on testing. According to this test, the NFHF performed nearly the same or slightly more corrosive than the base fluid (Table 6).

The MIL-H-46170 specification requires that these fluids meet the Federal Method 5322.1 requirements and pass after 10 days of exposure. The NFHF did not prevent galvanic corrosion when tested according to the Federal Method 5322.1 when exposed the required 10 days. However, NFHF did prevent galvanic corrosion by this method for a maximum of 6 days of exposure (Table 5).

MIL-H-46170 requires all three plates to pass when tested according to ASTM D1748. Only two out of three plates passed when tested according to ASTM D1748 using NFHF (Table 6).

SECTION IV. CONCLUSIONS

This evaluation reveals that the antiwear additive in the NFHF has improved the lubricity properties over CTFE base fluid and is equal to the MIL-H-46170 lubricity at higher loads using the ASTM D1472 laboratory test.

The corrosion inhibitor improved the performance of the NFHF over the CTFE base fluid in the ASTM D1748 (Humidity Cabinet) test and the Federal Test Standard Method 5322.1 (Bimetallic Couple Test). The NFHF was slightly more corrosive than the CTFE base fluid, but well within the MIL-H-46170 limits for weight change for the Federal Test Standard Method 5308.6 (Five Metal Coupon Test).

The physical and chemical characteristics of the NFHF are totally different from the currently-used synthetic hydrocarbon base fluid, MIL-H-46170. As a result, the comparison of the laboratory data of the two fluids may not be indicative of the true field performance of the NFHF since the existing requirements were established with petroleum-based fluids. The Air Force has completed field performance tests—as well as extensive laboratory tests—that indicate the corrosivity and lubricity properties of the NFHF are not factors for their applications. Based on the Air Force's results and the Army's research, it is evident that the NFHF would be suitable for use in future armored vehicles. Field performance evaluation of NFHF for Army applications should be the last phase of testing before considering adoption of the fluid for the future armored family of vehicles.

**Table 1. Wear Preventative Characteristics of Lubricating Fluid,
ASTM D 4172 on NFHF**

| WEAR SCAR DIAMETER (MM)* | | | | |
|--------------------------|-----------|-----------|-----------|----------------------|
| Run # | 10kg load | 15kg load | 40kg load | |
| | | | Maximum | Minimum |
| 1 | 0.5 | 0.444 | 0.636 | 0.558 |
| | 0.408 | 0.52 | 0.576 | 0.492 |
| | 0.524 | 0.464 | 0.596 | 0.518 |
| 2 | 0.54 | 0.468 | 0.646 | 0.546 |
| | 0.492 | 0.444 | 0.618 | 0.542 |
| | 0.492 | 0.464 | 0.622 | 0.532 |
| 3 | 0.58 | 0.452 | 0.682 | 0.516 |
| | 0.6 | 0.5 | 0.664 | 0.538 |
| | 0.508 | 0.484 | 0.64 | 0.516 |
| 4 | 0.492 | 0.524 | 0.618 | 0.522 |
| | 0.408 | 0.468 | 0.624 | 0.52 |
| | 0.56 | 0.496 | 0.652 | 0.516 |
| 5 | 0.448 | 0.408 | 0.676 | 0.568 |
| | 0.496 | 0.34 | 0.684 | 0.57 |
| | 0.52 | 0.42 | 0.678 | 0.54 |
| 6 | 0.476 | 0.504 | 0.608 | 0.506 |
| | 0.504 | 0.46 | 0.624 | 0.534 |
| | 0.496 | 0.504 | 0.622 | 0.53 |
| 7 | 0.524 | 0.416 | 0.614 | 0.494 |
| | 0.512 | 0.416 | 0.648 | 0.5 |
| | 0.476 | 0.4 | 0.626 | 0.496 |
| 8 | 0.576 | 0.456 | 0.624 | 0.54 |
| | 0.464 | 0.492 | 0.638 | 0.516 |
| | 0.472 | 0.512 | 0.642 | 0.534 |
| 9 | 0.508 | 0.472 | 0.674 | 0.572 |
| | 0.464 | 0.492 | 0.614 | 0.486 |
| | 0.408 | 0.468 | 0.618 | 0.528 |
| 10 | 0.464 | 0.456 | 0.69 | 0.652 |
| | 0.548 | 0.492 | 0.684 | 0.51 |
| | 0.408 | 0.44 | 0.694 | 0.47 |
| AVERAGE | 0.496 | 0.463 | 0.585 | (all min-max values) |
| STANDARD DEVIATION | 0.047 | 0.041 | 0.065 | (all min-max values) |
| MIN VALUE | 0.408 | 0.34 | 0.576 | 0.47 |
| MAX VALUE | 0.6 | 0.524 | 0.694 | 0.652 |

* All measurements were made on the Scherr Tumico optical comparator with the autometronics microprocessor.

**Table 2a. Corrosiveness and Oxidation Stability of Light Oils, Method 5308.6
(FTMS 791C) on NFHF**

| METAL WEIGHT CHANGE (MG/CM)* | | | | | |
|------------------------------|----------|---------|--------|---------|-----------|
| Run # | Aluminum | Cadmium | Copper | Iron | Magnesium |
| 1A | -0.008 | -0.008 | -0.184 | -0.032 | + 0.008 |
| B | -0.168 | -0.152 | -0.272 | -0.096 | -0.152 |
| 2A | -0.144 | - 0.2 | -0.232 | 0 | -0.024 |
| B | -0.104 | - 0.12 | - 0.28 | -0.088 | -0.104 |
| 3A | -0.008 | -0.032 | -0.168 | -0.008 | 0 |
| B | -0.016 | -0.056 | -0.232 | -0.048 | -0.032 |
| 4A | 0 | + 0.016 | -0.224 | + 0.056 | -0.024 |
| B | 0 | -0.016 | - 0.08 | + 0.024 | + 0.016 |
| 5A | + 0.05 | + 0.03 | -0.015 | 0 | + 0.06 |
| B | + 0.03 | + 0.04 | - 0.16 | + 0.05 | + 0.06 |
| 6A | -0.048 | - 0.04 | -0.232 | -0.048 | - 0.04 |
| 7A | + 0.008 | -0.016 | -0.168 | -0.016 | + 0.016 |
| B | + 0.032 | + 0.048 | -0.096 | + 0.048 | + 0.048 |
| C | -0.008 | -0.032 | -0.096 | + 0.016 | -0.008 |
| 8A | -0.008 | 0 | - 0.21 | -0.016 | -0.016 |
| B | + 0.008 | + 0.008 | - 0.16 | + 0.004 | + 0.048 |
| C | + 0.032 | + 0.024 | -0.128 | + 0.016 | + 0.016 |
| 9A | -0.016 | - 0.04 | -0.184 | -0.024 | -0.032 |
| B | -0.072 | + 0.072 | - 0.2 | -0.008 | + 0.064 |
| C | + 0.056 | + 0.048 | -0.104 | + 0.064 | + 0.072 |
| AVERAGE** | 0.029 | 0.033 | 0.158 | 0.026 | 0.036 |
| STANDARD DEVIATION** | 0.023 | 0.021 | 0.05 | 0.02 | 0.022 |
| MIN VALUE** | 0.008 | 0 | 0.096 | 0.004 | 0.008 |
| MAX VALUE** | 0.072 | 0.072 | 0.232 | 0.064 | 0.072 |

* The average change in total acid number (TAN) = 0.82.

** Includes only runs 6-9 with Teflon thread.

**Table 2b. Corrosiveness and Oxidation Stability of Light Oils, Method 5308.6
(FTMS 791C) on NFHF**

| RUN % | EVAPORATION % | VISCOSITY (cs)* | MODIFICATION | REMARKS |
|-----------------------------|---------------|-----------------|--------------|---|
| 1A | 9 | n/a | none | Cotton degrades |
| B | 69 | n/a | none | Leak; therefore, evaporation occurred |
| 2A | 3.01 | 3.1 | none | Cotton degrades |
| B | 6.4 | 3.2 | none | Cotton degrades |
| 3A | 8.76 | 3.3 | none | Cotton degrades |
| B | 4.99 | 3.2 | none | Cotton degrades |
| 4A | 12.31 | n/a | none | Cotton degrades |
| B | 5.55 | n/a | none | Cotton degrades |
| 5A | 7.33 | n/a | nylon | Absorbs corrosion preventative additive |
| B | 4.58 | n/a | nylon | |
| 6A | 7.95 | 3.1 | teflon | No complications |
| 7A | 8.97 | 3.1 | teflon | No complications |
| B | 8.03 | 3.1 | teflon | No complications |
| C | 6.93 | 3.1 | teflon | No complications |
| 8A | 5.21 | 3.1 | teflon | No complications |
| B | 5.72 | 3.1 | teflon | No complications |
| C | 5.57 | 3.2 | teflon | No complications |
| 9A | 5.38 | 3.1 | teflon | No complications |
| B | 7.03 | 3.1 | teflon | No complications |
| C | 5.38 | 3.1 | teflon | No complications |
| AVERAGE** | 6.62 | 3.1 | | |
| STANDARD DEVIATION** | 1.35 | 0.032 | | |
| MIN VALUE** | 5.21 | 3.1 | | |
| MAX VALUE** | 8.97 | 3.2 | | |

* The kinematic viscosity of NFHF is 3.1 cs at 38°C.

** Includes only runs 6-9 with teflon thread.

**Table 3. Corrosiveness of Oil on a Bimetallic Couple, Method 5322.1
(FTMS 791C) on NFHF**

| RUN # | NO. OF DAYS | NUMBER OF DISKS | | FINAL RESULTS |
|-------|----------------|--------------------------|-----------------------|------------------|
| | | No Signs of Corrosion | Signs of Corrosion | |
| 1 * | 10 | 3 | 0 | PASS |
| 2 | 10 | 7 | 3 | FAIL |
| 3 | 14 | 4 | 6 | FAIL |
| 4 | 20 | 5 | 15 | FAIL |
| 6 | 5 | 5 | 0 | PASS |
| | 6 | 5 | 0 | PASS |
| | 7 | 4 | 6 | FAIL |

The 50% humidity was maintained with H₂SO₄ due to a shortage of MgNO₂·6H₂O.

**Table 4. Rust Protection by Metal Preservatives in the Humidity Cabinet,
ASTM D1748* on NFHF**

| | PASS | FAIL |
|-----------------------|------|------|
| 12 polished plates | 8 | 4 |
| 12 sandblasted plates | 8 | 4 |
| 12 polished plates | 12 | 0 |
| 12 polished plates | 9 | 3 |
| 3 polished plates | 0 | 3 |
| 3 polished plates | 2 | 1 |
| 3 polished plates | 2 | 1 |

* 48.9°C for 100 hours.

Table 5. Proposed Corrosion Rate Evaluation Procedure (CREP) on NFHF

METAL WEIGHT CHANGE (MG/HR)

| Run # | Sandblasted | Polished |
|---------------------------|--------------------|-----------------|
| 1 | -1.2 | -0.9 |
| 2 | -1.1 | -1.5 |
| 3 | +0.5 | +0.3 |
| 4 | 0 | -0.1 |
| 5 | -0.7 | -0.8 |
| 6 | +0.8 | +0.7 |
| 7 | -0.8 | 0 |
| 8 | -0.1 | 0 |
| 9 | -1.6 | -0.8 |
| 10 | -0.4 | 0 |
| AVERAGE | 0.7 | 0.5 |
| STANDARD DEVIATION | 0.4 | 0.5 |
| MIN VALUE | 0 | 0 |
| MAX VALUE | 1.6 | 1.5 |

Table 6. Comparison of Wear and Corrosion Properties

FOUR-BALL WEAR ASTM D1472

| | Wear Scar (mm) | | |
|---------------------------------------|----------------|-----------|-----------|
| | 10kg load | 15kg load | 40kg load |
| CTFE base fluid ⁸ | 0.81 | 0.38 | 0.82-2.6 |
| Formulated NFHF | 0.50 | 0.46 | 0.47-0.69 |
| MIL-H-46170 Limits (max) ⁹ | 0.25 | 0.30 | 0.65 |

CORROSIVENESS, METAL STRIP FEDERAL METHOD 5308.6

| | TAN* Change | Al | Metal Weight Change (mg/cm) | | | |
|--|----------------|------|-----------------------------|------|------|------|
| | | | Cd | Cu | Fe | Mg |
| CTFE base fluid ⁸ | 0.0 | 0.00 | 0.00 | 0.03 | 0.00 | 0.00 |
| Formulated NFHF | 0.82 | 0.03 | 0.03 | 0.16 | 0.03 | 0.04 |
| MIL-H-46170 Limits (max) ⁹ | 0.30 | 0.2 | 0.2 | 0.6 | 0.2 | 0.2 |

CORROSION BIMETALLIC COUPLE, FEDERAL METHOD 5322.1

Exposure 10 days

| | |
|---------------------------------------|---------|
| Formulated NFHF | Fails** |
| MIL-H-46170 Limits (max) ⁹ | Passes |

CORROSION HUMIDITY CABINET, ASTM D1748

| | |
|---------------------------------------|------------------------|
| CTFE base fluid ⁸ | all 3 plates Fail*** |
| Formulated NFHF | 2 out of 3 plates Pass |
| MIL-H-46170 Limits (max) ⁹ | all 3 plates Pass |

PROPOSED CREP TEST

Metal Weight Change (mg/hr)

| | |
|-------------------------------|-----------|
| CTFE base fluid ¹⁰ | 1.5 - 2.5 |
| Formulated NFHF | 0.0 - 1.5 |
| MIL-H-46170**** | 0.0 -1.4 |

- * NFHF Total Acid Number (TAN) = 0.068.
- MIL-H-46170 TAN max = 0.20.

** Pass up to 6 days.

*** In 24 hours.

**** CREP is not a MIL-H-46170 requirement. Data generated in-house.

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